



Strengthening of nickel-based hybrid composites processed by spark plasma sintering, high-pressure torsion, and post-deformation annealing

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ABSTRACT

Nickel-based composites reinforced with nanosized and hybrid micro-/nanosized silicon carbide particles were fabricated using a processing route combining high-energy ball milling, spark plasma sintering (SPS), high-pressure torsion (HPT), and post-deformation annealing. The aim of this study was to establish the relationships between processing, microstructural evolution, and mechanical performance in Ni-SiC composites subjected to severe plastic deformation and subsequent heat treatment. Spark plasma sintering produced highly densified composites with a homogeneous distribution of the ceramic reinforcement and a refined nickel matrix. Detailed microstructural characterization revealed significant chemical and morphological evolution of the SiC reinforcement during SPS, including the formation of carbon-enriched shells around micro-sized particles and Ni-Si reaction products associated with nanosized reinforcement. High-pressure torsion resulted in a homogeneous ultrafine-grained microstructure accompanied by extensive grain refinement, fragmentation of micro-sized SiC particles, and redistribution of nanosized reinforcement throughout the matrix. These microstructural changes produced a substantial increase in hardness and tensile strength, but at the expense of tensile ductility after HPT. The Ni-hybridSiC composite achieved a tensile strength of 1526 MPa and an average microhardness of 720 HV after annealing at 300 °C, while its ultimate elongation remained approximately 0.59%. Thus, the principal mechanical benefit of the applied processing route is the development of ultrahigh strength and hardness

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rather than a balanced strength–ductility response. Among all investigated materials, the hybrid micro-/nano-SiC composite exhibited the most effective strengthening response, demonstrating the synergistic effect of bimodal reinforcement and severe plastic deformation on the development of ultrahigh-strength nickel-based composites.

Introduction

Nickel-based composites constitute a class of advanced structural and functional materials in which a nickel or nickel-alloy matrix is reinforced with ceramic particles [1], solid lubricants [2], carbon nanostructures [3], or intermetallic phases [4] to enhance performance in severe mechanical, thermal, and corrosive environments. Nickel is selected as the matrix due to its high melting point, excellent oxidation and corrosion resistance, and strong mechanical stability at elevated temperatures. Furthermore, its electrochemical properties make it an attractive candidate for sustainable energy storage and conversion systems [5]. The incorporation of reinforcements such as Al_2O_3 [6], SiC [7], WC [8], TiC [9], carbon nanotubes [10], graphene nanoplatelets [11], or NiAl intermetallics [12] enables tailored microstructures and multifunctional behavior. The improved mechanical performance of Ni-based composites arises from several strengthening mechanisms, including load transfer to stiff reinforcements, Orowan looping, dislocation pinning, grain refinement, and thermal expansion mismatch strengthening. As a result, these materials exhibit increased hardness, yield strength, elastic modulus, fatigue resistance, and creep resistance, particularly at elevated temperatures [13, 14]. Owing to their combination of superior performance, Ni-based composites are excellent candidates for aerospace and energy systems, automotive applications, chemical and marine equipment, and advanced tribological systems [15].

A wide range of processing techniques is employed to fabricate Ni-based composites, including powder metallurgy routes [16], electrodeposition for protective coatings [17], thermal spraying [18], and additive manufacturing techniques [19]. Recently, the most applicable method for manufacturing bulk Ni-based composites is spark plasma sintering (SPS), which uses an electric current and external pressure to densify the powder mixture [20–23]. The spark plasma sintering technique significantly reduces processing time and improves energy efficiency compared with conventional sintering routes [24]. Its principal advantages

include enhanced microstructural control and grain-growth suppression, resulting from the lower sintering temperatures employed. These features are critically important for materials processing, particularly in the fabrication of nanocomposites, by promoting effective surface purification and activation of powder particles, facilitating diffusion bonding, and facilitating densification [25].

A special group of nickel-based composites is the one reinforced with SiC particles. Nickel–silicon carbide spark plasma-sintered composites exhibit significant improvements in mechanical properties due to controlled interfacial reactions at the matrix–reinforcement interface. Recent studies of SPS Ni–SiC composites have revealed pronounced microstructural evolution, driven by an intense reaction between nickel and silicon, which results in the partial or complete dissolution of silicon carbide [16]. Consequently, a multicomponent interphase developed, consisting of a nickel silicide matrix ($\text{Ni}_{31}\text{Si}_{12}$ and/or Ni_3Si) enriched with carbon nanoprecipitates [7]. With increasing thermal exposure, these nanoprecipitates coarsened and agglomerated into carbon particles, which subsequently segregated at the interface between the nickel matrix and the interphase. The application of ultralow SiC additions (0.001 wt%) resulted in satisfactory strengthening, achieving a 115% increase in flexural strength at 20 °C (1130 MPa) and 86% at 400 °C (976 MPa) compared to pure nickel [26]. Moreover, 1.5 wt% SiC composites showed superior combined strength and plasticity compared with pure nickel, confirming the ability to produce dense, strengthened Ni–SiC composites through dispersion- and Hall–Petch-strengthening mechanisms. However, such strengthening comes at the cost of decreased ductility, which can be further limited by interfacial porosity [27]. The same authors analyzed the effect of mixing/milling time on the mechanical properties of SPS Ni–SiC nanocomposites at room temperature and at elevated temperatures [28]. They showed that increasing the milling time (up to 48 h) of Ni–SiC composites refines the Ni matrix to an ultrafine-grained structure and enhances SiC dispersion, significantly increasing

tensile strength, though elongation decreases. This disadvantage has been partially mitigated by annealing as the final step in the fabrication process [29]. Low-temperature annealing effectively reduced dislocation density, resulting in a slight increase in total elongation while maintaining a relatively high tensile strength, owing to the preserved refined grain size.

Grain refinement of the metal matrix appears to be a fundamental factor in composite strengthening. Subsequently, research tries to exploit nano-reinforcement for further improvement. Nanoparticles are used as the sole reinforcement and in hybrids, where micro- and nanosized ceramics are used simultaneously. One route to combine a nano-grained matrix with nano-sized reinforcement in bulk form is powder metallurgy (such as Spark Plasma Sintering, SPS), followed by Severe Plastic Deformation (SPD). It represents an advanced processing route that enables near-full densification, refined microstructures, and superior mechanical and tribological performance [30, 31].

High-pressure torsion (HPT) is a severe plastic deformation method that induces exceptionally large shear strains, making it one of the most effective routes for producing ultrafine-grained structures and markedly increasing material strength [32]. During processing, the specimen is compressed under large quasi-hydrostatic pressure (several GPa) while undergoing simultaneous torsional rotation. After high-pressure torsion (HPT), the composite undergoes profound microstructural refinement and homogenization, indicating the formation of ultrafine grains, the redistribution of reinforcements, and enhanced interfacial integrity.

Recently, a few studies examined nickel-based composites processed by HPT. Carbon nanotube (CNT)-reinforced nickel composites exhibited a hardness increase of up to 800% relative to the sintered sample [33]. Similar work was reported in [34], where Nickel/CNT composites with varying CNT content were processed by HPT at different temperatures to optimize the process parameters. It was shown that the optimal HPT processing route to improve CNT distribution is a combination of deformation at 400 °C followed by deformation at room temperature. Also, nickel-graphene composites processed by HPT at 200 °C showed optimal strength and ductility, whereas those with 2% graphene at room temperature exhibited superior microhardness [35].

In summary, correlating the microstructural evolution of nickel-based composites processed by SPS

and subsequent HPT with their mechanical performance remains a highly relevant and actively investigated topic. However, the existing literature on HPT-processed Ni-based composites remains limited and fragmented, and there are no comprehensive studies of the microstructure–property relationships in Ni–SiC systems subjected to HPT. The presented research aims to fill this gap. It proposes an investigative methodology comprising a manufacturing route based on high-energy milling, SPS, HPT, and annealing of nickel–silicon carbide nano- and hybrid composites. The workflow of the experimental methodology is shown in Figure 1. The next section describes the research plan in detail.

Experimental methods and methodology

Manufacturing procedure

The manufacturing process consists of four main stages: high-energy mixing, spark plasma sintering, high-pressure torsion, and annealing (Figure 1). Firstly, to ensure a homogeneous distribution of constituents within the powder blend, high-energy mixing was carried out in a planetary ball mill (Fritsch Pulverisette 6). High-energy milling is widely used in metal matrix composite (MMC) processing to achieve uniform dispersion of reinforcement particles and enhanced contact between matrix and ceramic phases [36]. As a result, high-energy milling facilitates densification during consolidation and improves the mechanical performance and structural homogeneity of the final composite [37].

Nickel powder (NewMet Koch, 5 µm, 99.9%), micro-sized silicon carbide (Saint-Gobin, 80 µm, 99.99%), and nanosized silicon carbide (DorChem, particle size < 50 nm) were chosen as the matrix and hybrid reinforcement materials, respectively. The morphology of the initial powders is depicted in Figure 2. A nickel–SiC composite powder was prepared in a tungsten carbide/cobalt (WC/Co) container. The milling conditions were: rotational speed $\omega = 200$ rpm, duration $t = 40$ h, ball-to-powder ratio of 5:1, and 10 mm diameter milling balls. Two types of Ni–SiC composites were produced: first, with 3 vol.% content of nanosized SiC powder (denoted as *Ni-nanoSiC*), and second, with 1.5 vol.% nanosized + 1.5 vol.% micro-sized SiC powder (denoted as *Ni-hybridSiC*).

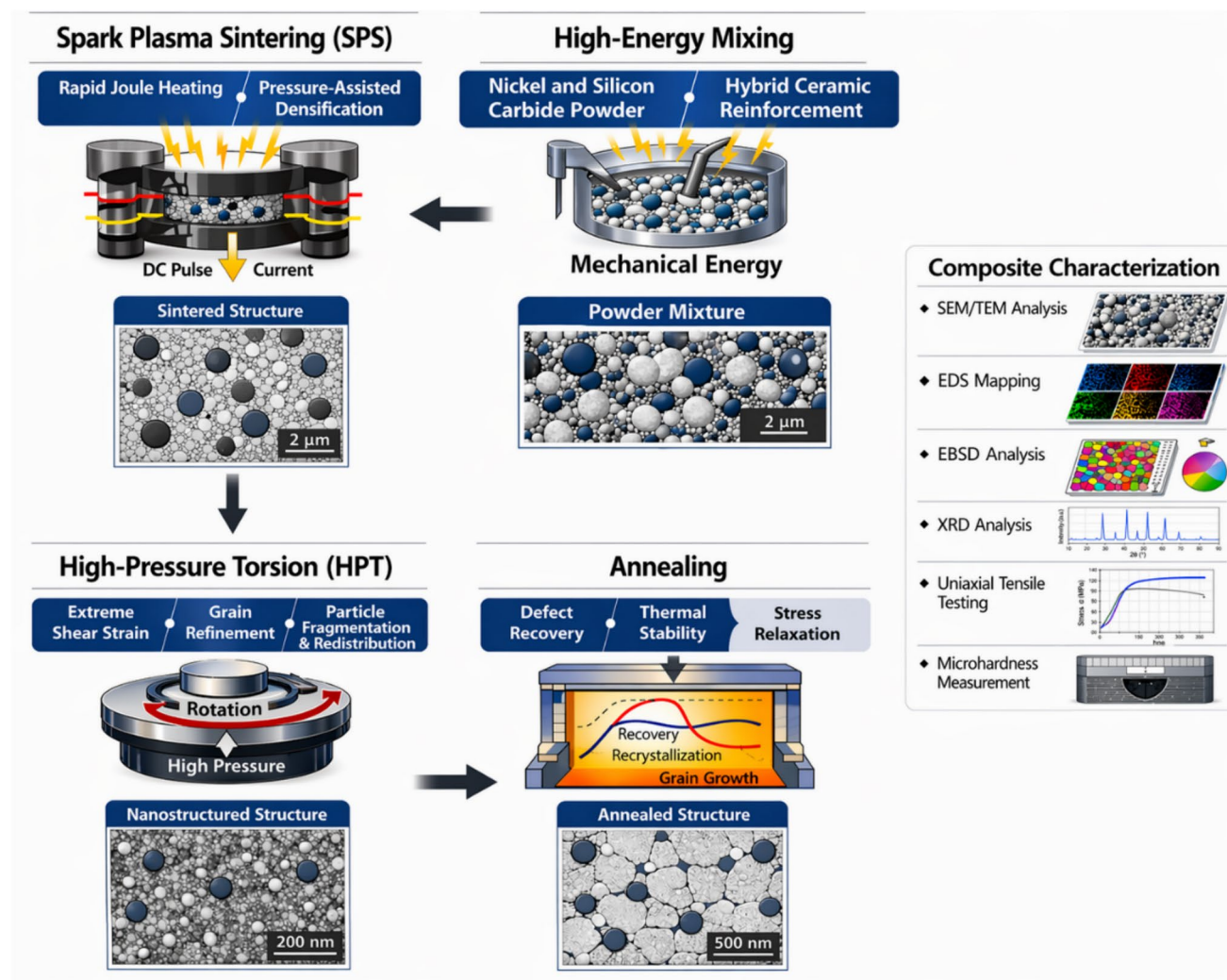


Figure 1 The workflow of the experimental methodology for processing and characterization of nickel-based hybrid composites.

Powder consolidation was performed by spark plasma sintering (SPS) with the following sintering parameters: sintering temperature = 1000 °C, sintering time = 10 min, and external pressure = 50 MPa. The sintering conditions were established based on previous investigations on Ni–SiC composite fabrication [7, 16]. The specimens were heated at 100 °C/min to 700 °C, then the heating rate was reduced to 50 °C/min until the target sintering temperature was reached. Following the dwell period, the applied pressure was released from 50 MPa to 0 MPa, and the samples were allowed to cool naturally to room temperature at an average rate of approximately 10 °C/min.

Following the initial sintering step, the specimens were processed by high-pressure torsion (HPT). Disks measuring 10 mm in diameter and 1 mm in thickness

were deformed under an applied pressure of 6 GPa, with the lower anvil rotating at 1.4 rpm for 10 revolutions at room temperature. The deformation was performed under quasi-constrained conditions, permitting limited material outflow at the disk periphery during processing.

Post-deformation annealing of HPT-processed composites was performed to relieve residual stresses and stabilize the highly strained microstructure [38]. Annealing was performed at 200, 300, 400, 500, 600, and 700 °C in air using a furnace preheated to the target temperature. The samples were introduced into the furnace after the target temperature was reached and held there for 1 h. Controlled heat treatment promotes recovery and partial recrystallization of the ultrafine grains while preserving the refined structure

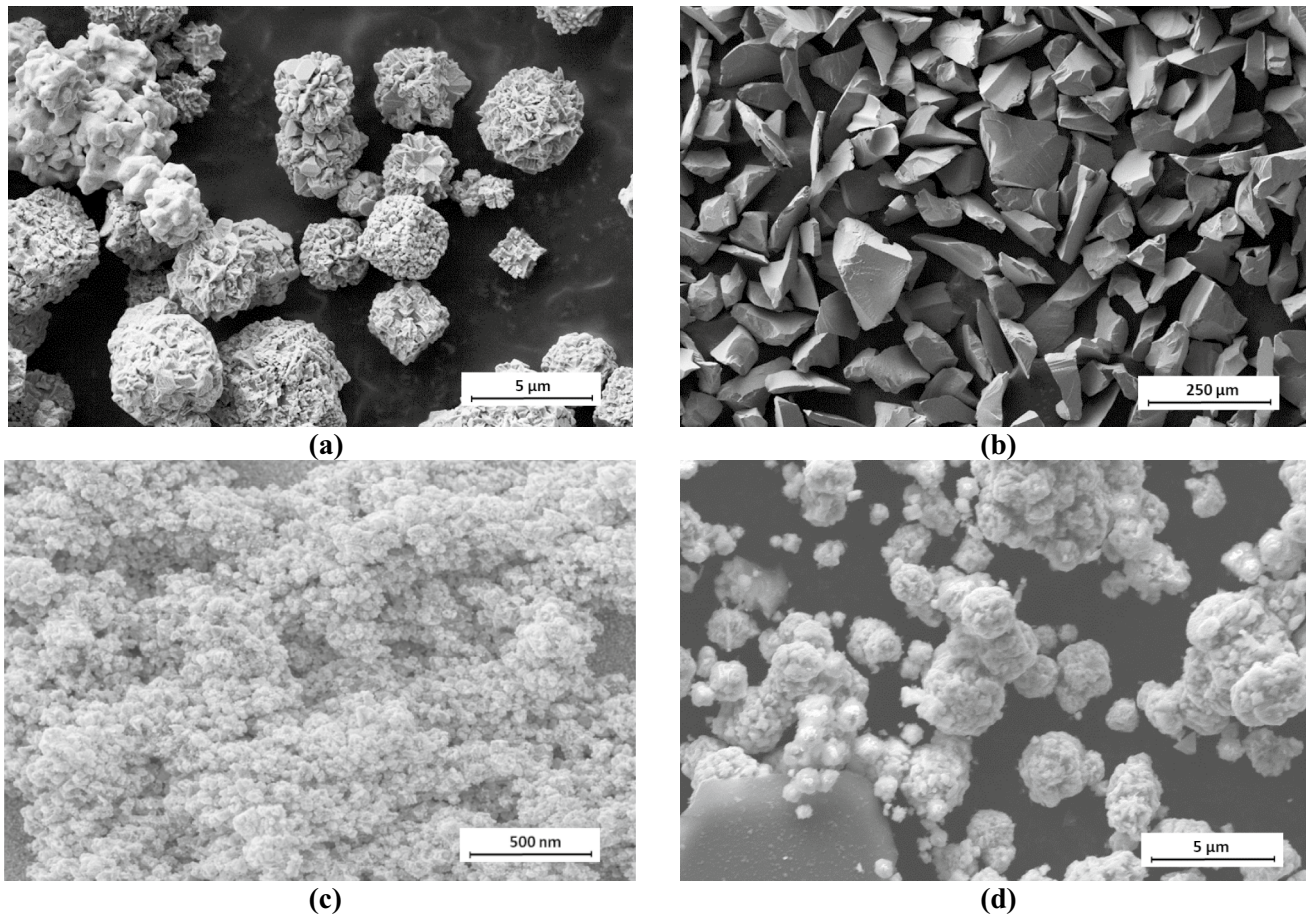


Figure 2 The morphology of applied powders: **a** Ni, **b** microsized SiC, **c** nanosized SiC, and **d** mixture of Ni-(1.5% vol. nano+1.5% vol. micro) SiC after high-energy milling.

generated during SPD processes. As shown, annealing can improve ductility and microstructural stability while maintaining a favorable balance between strength and toughness [39].

The as-processed materials were sectioned, ground, and polished to prepare specimens for subsequent characterization. The density of the Ni-SiC composites was measured using the hydrostatic method based on Archimedes' principle. The relative density (ρ_{rel}) was then calculated as the ratio of the measured bulk density (ρ_{m}) to the theoretical density (ρ_{t}) of the composite, assuming densities of $\rho_{\text{Ni}} = 8.9 \text{ g/cm}^3$ and $\rho_{\text{SiC}} = 3.2 \text{ g/cm}^3$, respectively.

Characterization procedure

The characterization of nickel composites was divided into microstructural and mechanical ones. Firstly, a scanning electron microscope (SEM, Hitachi SU-8000)

equipped with energy-dispersive spectroscopy (EDS) was used to analyze the morphology of Ni-SiC composites and their elemental content. Observations were made on material cross sections at an accelerating voltage of 10 kV. Images were taken in two modes. The secondary electron (SE) mode enabled surface analysis, while the backscattered electron (BSE) mode, with higher mass contrast, provided information about the phases and grain boundaries in the samples.

Secondly, electron backscatter diffraction (EBSD) was employed to investigate grain morphology after each of the main fabrication steps (SPS, HPT, and annealing). All samples were mechanically ground using SiC papers (grit sizes #600 and #1200), followed by polishing with 3 μm and 1 μm diamond suspensions. Final surface preparation was performed using vibropolishing with a 1 μm diamond suspension. After preparation, EBSD mapping was successfully performed for the SPS samples. However, for

HPT-deformed and annealed samples, the grain size was comparable to the resolution limit of conventional EBSD. Therefore, thin lamellae were prepared using focused ion beam (FIB) milling, following a standard lift-out procedure, for transmission Kikuchi diffraction (TKD) analysis at higher resolution. As a result, crystallographic orientation maps were successfully obtained for all processing conditions. The entire procedure was conducted on the SEM-FIB Zeiss Crossbeam 350 with an EDAX EBSD detector. For EBSD, an accelerating voltage of 20 kV was used, with a step size of 1 μm . For TKD, an off-axis geometry was used with an accelerating voltage of 20 kV and a step size of 10 nm. For both cases, grain boundaries were defined using a misorientation threshold of 15°.

X-ray diffractometry (XRD) was carried out using a Bruker D8 Discover in coupled scan mode, with Cu-K α radiation ($\lambda = 1.5418 \text{ \AA}$). Measurements were taken with a collimated beam due to the limited sample surface area (1 mm diameter collimator), thereby maintaining relatively high signal intensity from the material. The crystalline size was calculated using EVA 5.2 analysis software, with the Create Area tool. Based on the Scherrer evaluation, the full width at half height (FWHM) of the peaks was used to analyze them.

Mechanical properties were evaluated by Vickers microhardness measurements and uniaxial tensile testing. Hardness testing was performed using an INNOVATEST Europe BV FALCON 500 system equipped with a Vickers indenter. The specimens were sectioned transversely and prepared as metallographic cross sections by mechanical grinding with SiC papers (240–1200 grit) followed by polishing using a 1 μm diamond suspension. Indentations were made along the disk diameter on the cross section with a spacing of 0.3 mm between impressions, using a load of 100 g and a dwell time of 10 s. The hardness distribution was subsequently analyzed by plotting hardness versus distance from the disk center. The average microhardness reported was calculated as the arithmetic mean of the individual Vickers measurements obtained along the complete measured diameter of the specimen. This value provides a convenient global measure for comparison between processing conditions and compositions. However, because HPT produces a radial strain gradient, the average value does not represent the local mechanical state at every position. Therefore, the complete radial hardness profiles are used to assess local heterogeneity. Small differences in the maximum measured radial distance arise from limited peripheral

material outflow during quasi-constrained HPT and subsequent metallographic preparation; they do not correspond to different nominal HPT-tool radii.

Uniaxial tensile tests were conducted at room temperature using a Zwick/Roell Z005 testing machine under displacement control at an initial strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$. Miniature dog-bone-shaped specimens with an overall length of 8.6 mm, a gage length of 5 mm, a gage width of 0.8 mm, and a thickness of 0.6 mm were used. Five independent specimens were tested for each material and processing condition. For the HPT-processed materials, the specimens were machined across the diameter of the 10-mm HPT disks. Owing to their overall length of 8.6 mm, the tensile specimens extended across most of the disk diameter and therefore sampled a substantial portion of the radial microstructural and mechanical property gradient generated during HPT. Engineering stress–strain curves were obtained from recorded data, and engineering strain was determined using digital image correlation (DIC).

Results

Microstructure characterization

SPS samples

The SEM image in Figure 3a illustrates the microstructure of a spark plasma-sintered Ni–SiC hybrid composite reinforced with both micro- and nanoscale silicon carbide particles. The nickel matrix exhibits a well-defined polycrystalline structure with clearly visible grain boundaries and equiaxed grains, indicating limited grain growth during high-temperature SPS consolidation. Two distinct populations of reinforcement particles are visible. The micro-sized particles appear as relatively large, dark inclusions embedded within the Ni matrix and are distributed relatively homogeneously across the grain boundaries. These particles are typically several micrometers in size and exhibit continuous interfaces with the surrounding matrix, indicating good mechanical interlocking and limited interfacial reaction during sintering. The nanosized particles appear as fine, dark features distributed homogeneously within the matrix, particularly in the magnified inset. These nanoscale reinforcements are heterogeneously distributed, but with no clear sign of detrimental

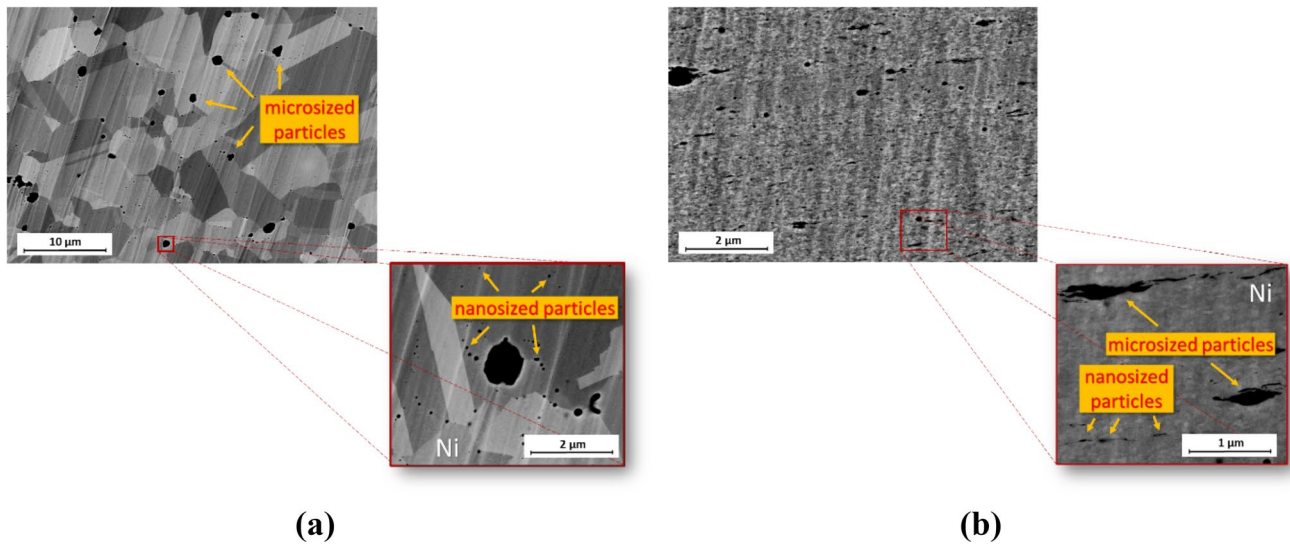


Figure 3 SEM images of Ni-hybridSiC after: **a** spark plasma sintering and **b** high-pressure torsion.

agglomerations. Nanoparticles can be observed in both grain boundaries and grain interiors, confirming the effectiveness of the high-energy milling process.

The effect of the milling process is also evident in Figure 4, where the EBSD maps reveal a clear transition in microstructure with increasing reinforcement addition. The pure Ni sample processed by

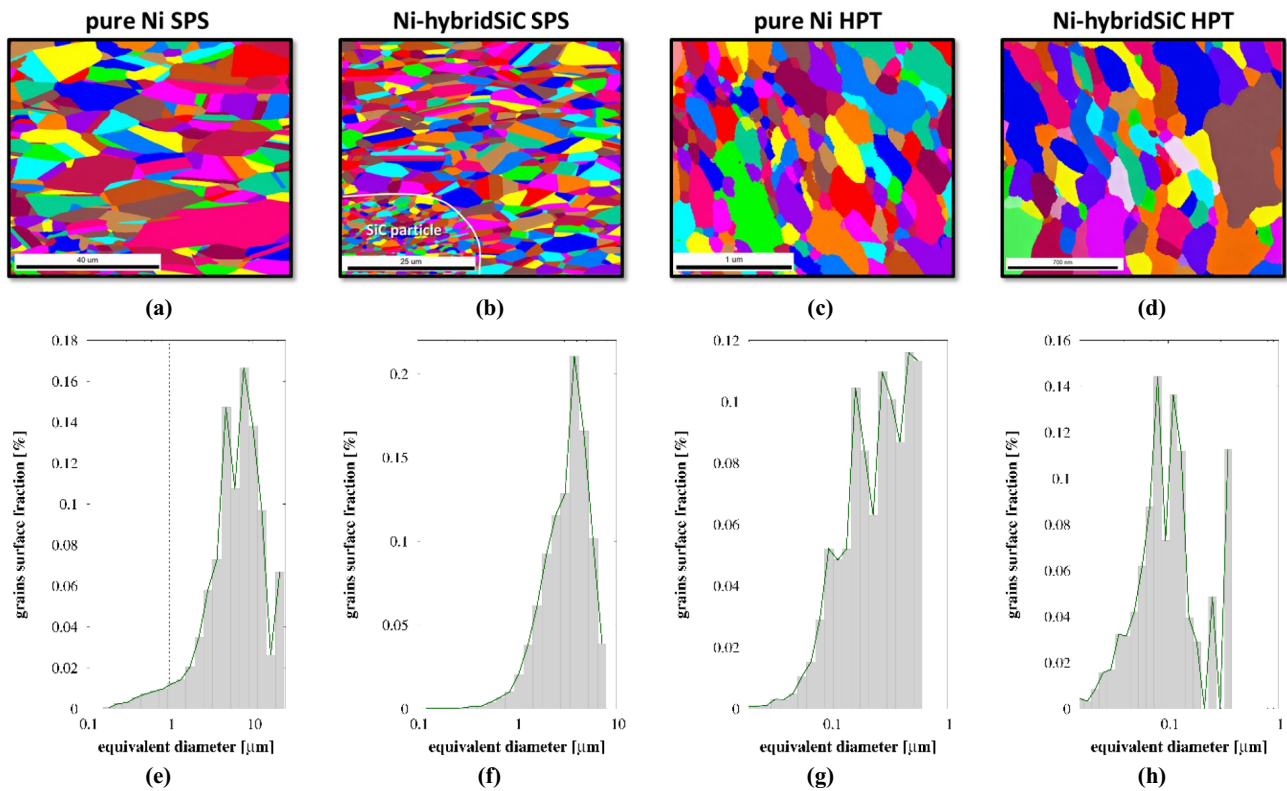


Figure 4 EBSD maps **a–d** and matrix grain size distributions **e–h** of SPSed/HPTed Ni and Ni-SiC samples.

SPS (Figure 4a) exhibits equiaxed grains with relatively large sizes and smooth boundaries, indicating that grain growth dominates during sintering. The Ni-hybridSiC SPS sample (Figure 4b) shows grain refinement and increased heterogeneity, with the visible part consisting of an agglomeration of small ceramic grains. The corresponding matrix grain size distributions (Figure 4e and f) confirm these observations, showing a progressive shift toward smaller equivalent diameters. The average grain area decreased from $7.97 \mu\text{m}^2$ to $3.48 \mu\text{m}^2$ upon addition of hybrid SiC reinforcement to the nickel matrix.

TEM analysis combined with EDS mapping confirms the heterogeneous reinforcement architecture of the Ni-hybridSiC composite (Figure 5). The investigated region contains a micro-sized particle surrounded by several finer particles, illustrating the coexistence of different reinforcement length scales characteristic of the hybrid composite design. A more detailed analysis of the micro-sized reinforcement particle reveals a distinct spatial separation between the Si and C signals. The EDS map shows that Si is predominantly concentrated in the particle core, whereas C is mainly enriched in the outer shell surrounding

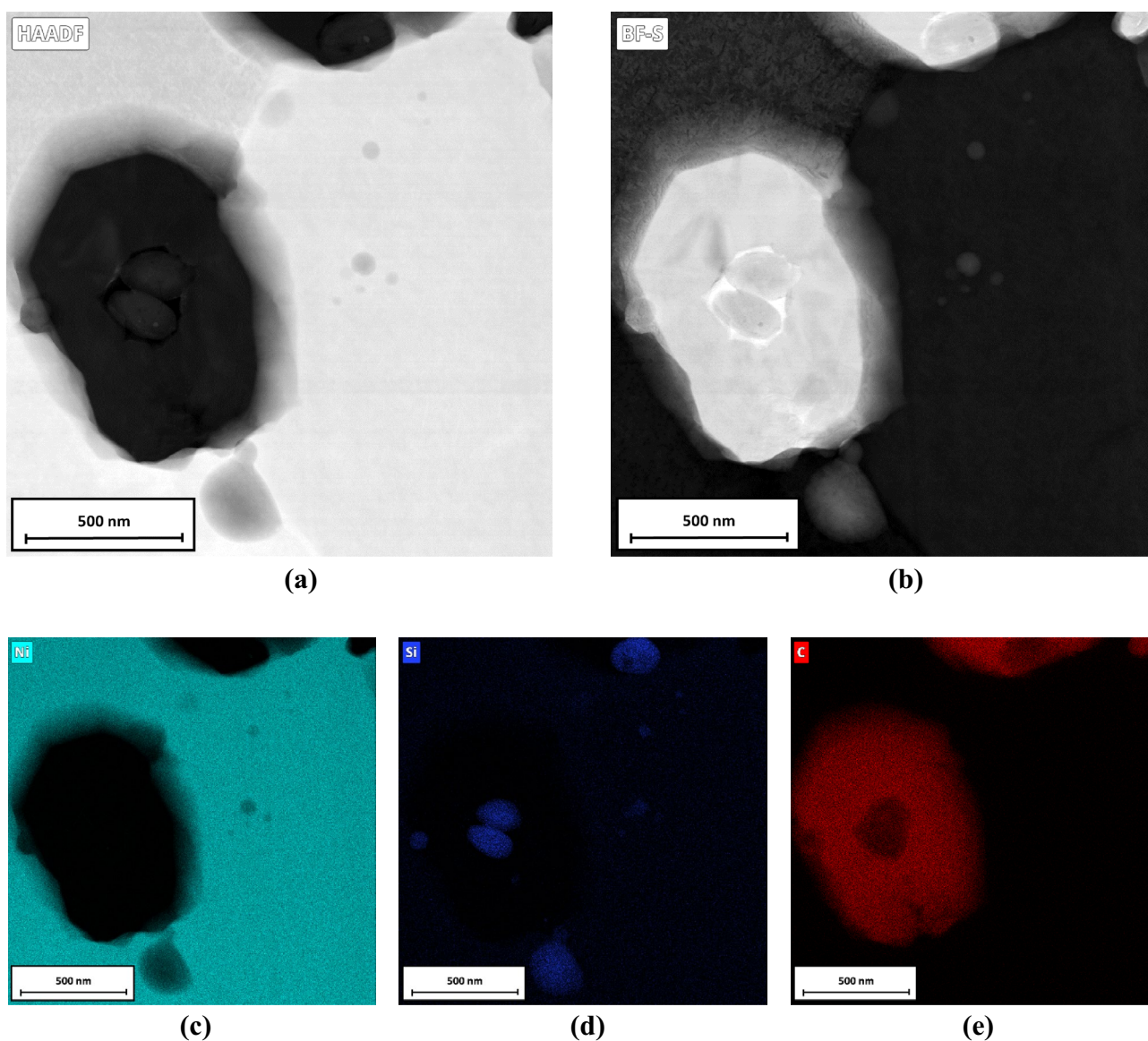


Figure 5 TEM images: HAADF **a** and BF-S **b** modes with EDS analysis: Ni **c**, Si **d**, and C **e** content, of Ni-hybridSiC after spark plasma sintering.

the Si-rich region. This indicates that the micro-sized particle does not exhibit a chemically uniform distribution of SiC. Still, rather a core-shell-like structure composed of a Si-rich interior and a C-rich peripheral layer. Such elemental partitioning may result from local decomposition, interfacial reactions, or redistribution of carbon during spark plasma sintering. The presence of a carbon-enriched shell at the particle-matrix interface may also influence interfacial bonding and load transfer between the ceramic reinforcement and the Ni matrix.

A detailed inspection of the nanosized particles located in the vicinity of the micro-sized reinforcement reveals the simultaneous presence of Si and Ni signals. The Si map confirms that these nanoscale features are enriched in silicon, indicating their association with the ceramic reinforcement phase. However, the Ni map also shows a detectable signal within the same regions, suggesting possible chemical interaction at the particle-matrix interface.

HPT samples

The microstructure of the Ni-SiC composite processed by high-pressure torsion (HPT) is shown in Figure 3b. It reveals a highly refined, anisotropic microstructure governed by intense shear deformation. The Ni matrix exhibits a lamellar and banded morphology aligned with the shear direction, indicating severe plastic strain localization and accumulation of lattice defects. The grains of the Ni matrix correspond to an ultrafine-grained structure. The mixture of elongated grains and ultrafine, equiaxed fragments, suggesting enhanced fragmentation, is shown in Figure 4c and d. In both pure Ni and Ni-hybridSiC conditions, HPT induces intense shear deformation, resulting in grains aligned along the deformation direction and a significantly increased grain-boundary density. The corresponding matrix grain size distributions confirm this observation, showing a substantial shift toward sub-micron-equivalent diameters in the HPT samples (Figure 4g and h) relative to the micrometer-scale grains observed after SPS. This refinement is quantitatively reflected in the average grain area, which decreases from $7.97 \mu\text{m}^2$ (Ni SPS) to $0.28 \mu\text{m}^2$ (Ni HPT), and from $3.48 \mu\text{m}^2$ (Ni-hybridSiC SPS) to $0.13 \mu\text{m}^2$ (Ni-hybridSiC HPT). In particular, the HPT-processed materials exhibit narrower distributions with peaks located at much smaller grain sizes, indicating a more uniform

ultrafine-grained structure. The presence of hybrid SiC particles further enhances refinement during HPT, resulting in the lowest average grain area and confirming the synergistic effect of particle reinforcement and severe plastic deformation. The SiC reinforcement exhibits a bimodal size distribution, confirming the composite's hybrid character. The nano-SiC particles are finely dispersed within the matrix and along grain boundaries, with an average size of approximately 50 nm. In contrast, the initially larger micro-sized particles appear fragmented and elongated in the shear direction, with an average equivalent size of approximately $0.5 \mu\text{m}$.

The conclusions from the SEM and EBSD investigations of the Ni-hybridSiC composite after high-pressure torsion have been confirmed by TEM analysis (Figure 6). The HAADF image shows elongated, high-contrast features embedded within a relatively uniform Ni matrix, indicating the presence of fragmented SiC particles aligned along the shear direction. The corresponding BF-STEM image confirms the existence of lamellar structures, reflecting intense strain localization during HPT processing. EDS elemental mapping demonstrates a homogeneous distribution of Ni throughout the matrix and, in part, within regions of nanosized particles. The Si and C maps reveal a heterogeneous distribution of the reinforcing phase. Notably, two distinct populations of SiC-derived particles can be identified. The larger, elongated particles exhibit a relatively stronger carbon signal, suggesting local carbon enrichment, partial decomposition, or interfacial reactions. Finally, the smaller, more equiaxed particles exhibit a pronounced co-localization of Si and Ni signals, suggesting that the original nanosized reinforcement particles reacted with the Ni matrix, forming a Ni-Si reaction regions.

Characterizing nanostructured HPT samples with EBSD is challenging because the technique's limited spatial resolution is insufficient for the extremely small grain sizes [40]. The very fine grains hinder accurate indexing and reliable determination of crystallographic orientations, potentially leading to an incomplete or biased representation of the true microstructure. As a result, the initially acquired EBSD maps often contain a high fraction of non-indexed (unresolved) pixels [41]. Post-processing approaches, such as data cleaning, interpolation, smoothing, and/or reconstruction algorithms, can be applied to improve map continuity and visualization quality [42–44] (see Figure 4c and d). However,

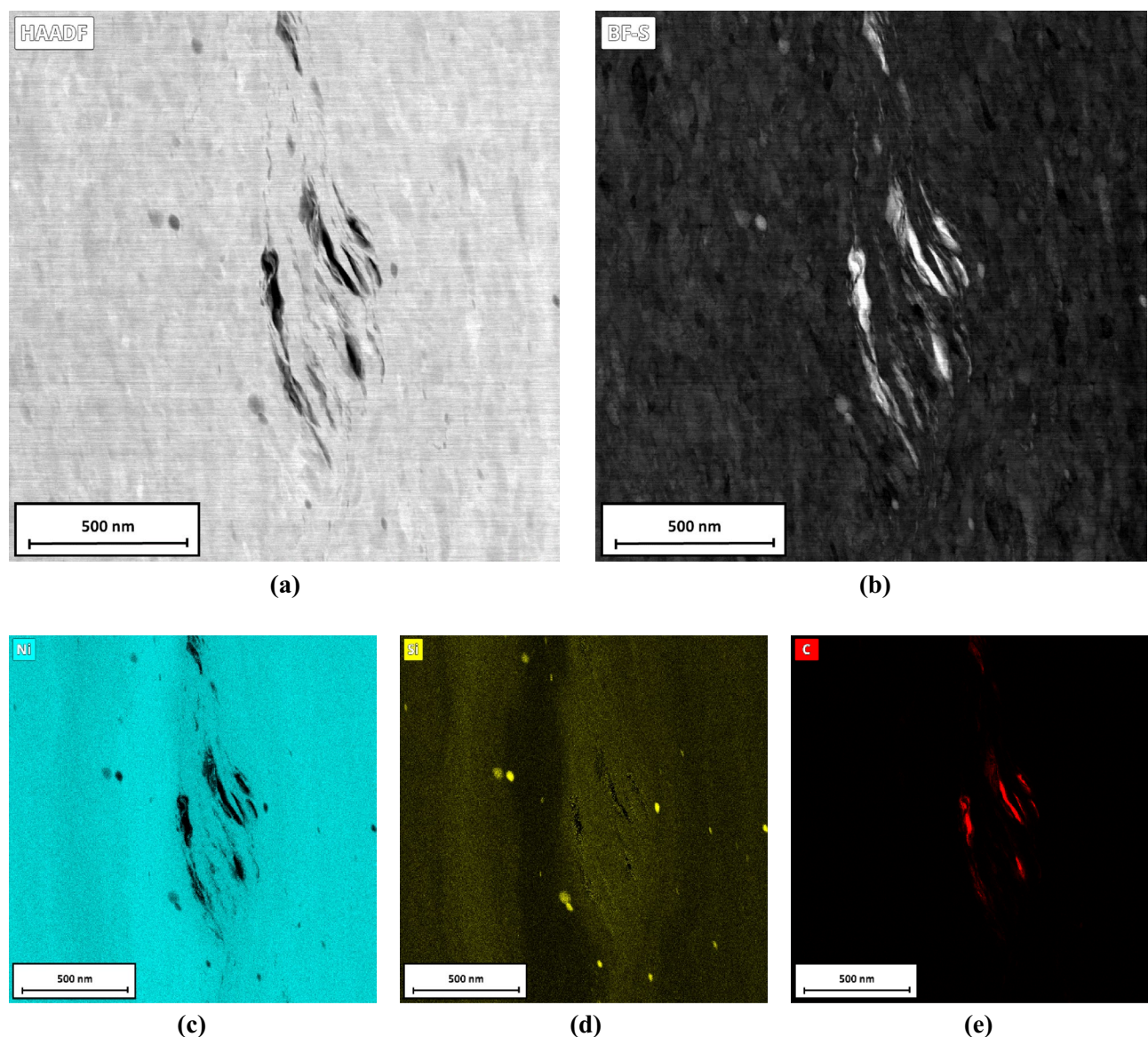


Figure 6 TEM images: HAADF **a** and BF-S **b** modes with EDS analysis: Ni **c**, Si **d**, and C **e** content, of Ni-hybridSiC after high-pressure torsion.

these procedures inherently involve spatial averaging, which may locally distort grain boundaries or artificially modify grain morphology. Consequently, while such treatments enhance interpretability, they must be applied with caution, as they can introduce discrepancies between the processed EBSD maps and the actual nanostructure.

To overcome the limitations of EBSD in HPT nanostructures, XRD was used to determine the crystallite size of the Ni matrix. The XRD patterns of the HPT-processed samples exhibit characteristic diffraction peaks corresponding to the FCC structure of nickel,

with no additional (SiC-derived) phases detected (Figure 7a). A noticeable broadening of the diffraction peaks is observed for the Ni-hybridSiC HPT sample compared to pure Ni, indicating a reduction in crystallite size induced by severe plastic deformation. The Scherrer analysis confirms this trend, showing smaller crystallite sizes in the composite than in unreinforced Ni, reflecting enhanced fragmentation mechanisms promoted by the presence of SiC particles (Figure 7b). Upon annealing at 300 °C and 400 °C, a gradual sharpening of the peaks is observed, corresponding to an increase in crystallite size due to recovery and

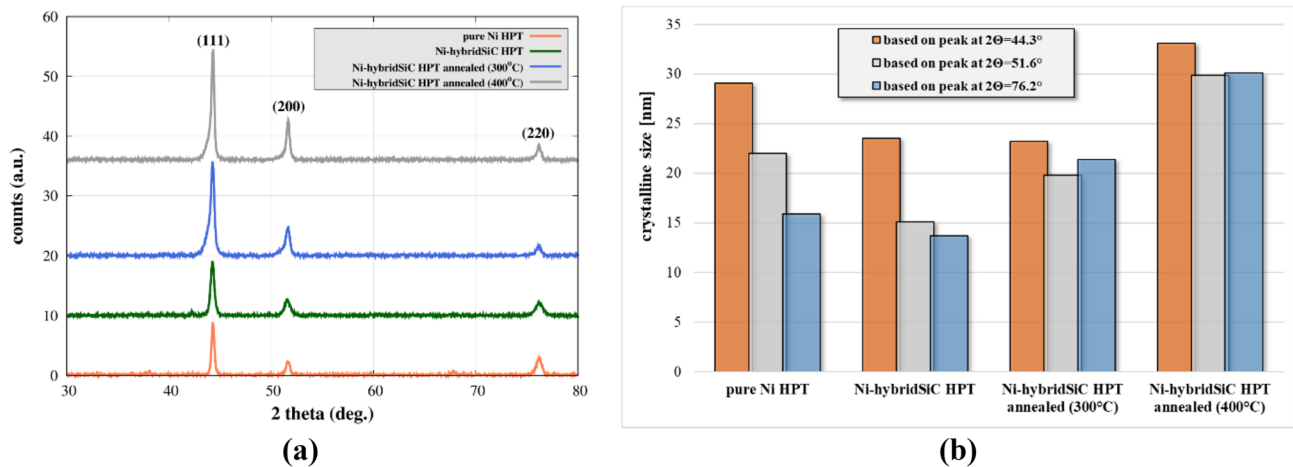


Figure 7 XRD analysis of crystalline size: **a** XRD patterns and **b** Scherrer evaluation.

partial recrystallization processes. The crystallite size increases more significantly at 400 °C, indicating intensified grain coarsening at elevated temperatures. In conclusion, the results demonstrate that adding SiC particles enhances refinement efficiency during HPT. At the same time, subsequent annealing induces controlled microstructural coarsening, which should influence the effective mechanical behavior of the composites.

Mechanical properties

Based on the methodology presented in "Experimental methods and methodology" section, a comprehensive analysis of the mechanical properties of SPS, HPT, and annealed samples was conducted. Microhardness, tensile strength, ultimate elongation, and failure elongation were determined for the Ni and Ni-SiC samples. The results are presented in Table 2 and Figure 8 as average values with standard error. Each mechanical property was studied and is discussed in the following paragraphs.

Microhardness

The average microhardness results reveal a clear strengthening trend in Ni-SiC composites as a function of processing route. Samples consolidated by SPS exhibit relatively low hardness, with values of 108 HV for pure Ni, 150 ± 1 HV for Ni-nanoSiC, and 145 HV for Ni-hybridSiC, indicating only a modest reinforcement effect. The appearance of a significantly harder

phase in the material suggests that SiC particles have most likely contributed to dispersion strengthening by impeding dislocation motion. The application of high-pressure torsion (SPS + HPT) leads to a pronounced increase in average microhardness for all compositions, reaching 445 ± 2 HV for pure Ni, 523 ± 6 HV for Ni-nanoSiC, and 533 ± 9 HV for Ni-hybridSiC. This significant enhancement reflects intensive grain refinement and strain hardening induced by severe plastic deformation. The improvement is more pronounced in SiC-reinforced systems, confirming the contribution of particle strengthening and more efficient load transfer at the refined microstructural scale. The relatively small standard error values for SPS + HPT samples indicate relatively good repeatability and microstructural homogeneity after deformation processing.

The local microhardness distribution along the disk radius reveals a characteristic gradient associated with the strain heterogeneity introduced during HPT processing (Figure 9). For all HPT-processed samples, the lowest local microhardness is observed near the disk center (≈ 0 mm), where the imposed shear strain is lower. In comparison, local hardness progressively increases with radial distance from the disk center ($\approx \pm 4$ –5 mm), corresponding to higher accumulated strain and resulting in greater fragmentation of the microstructure. This trend is particularly pronounced for the Ni-hybridSiC composite, which exhibits a significant increase in local microhardness from ~ 420 –440 HV in the central region to ~ 580 –600 HV at the edges of the disc, where the imposed shear strain reaches its maximum. A similar, though slightly less

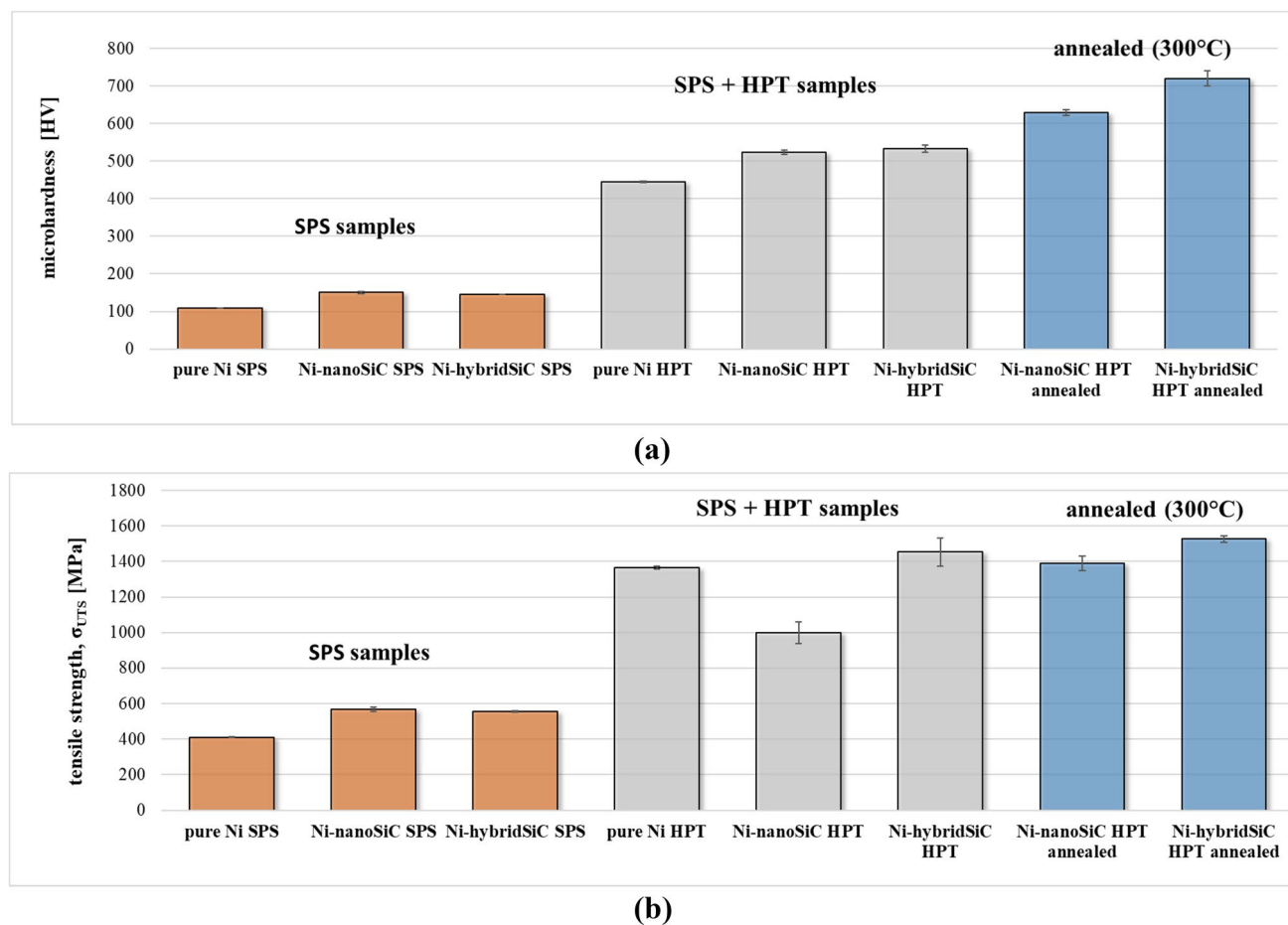
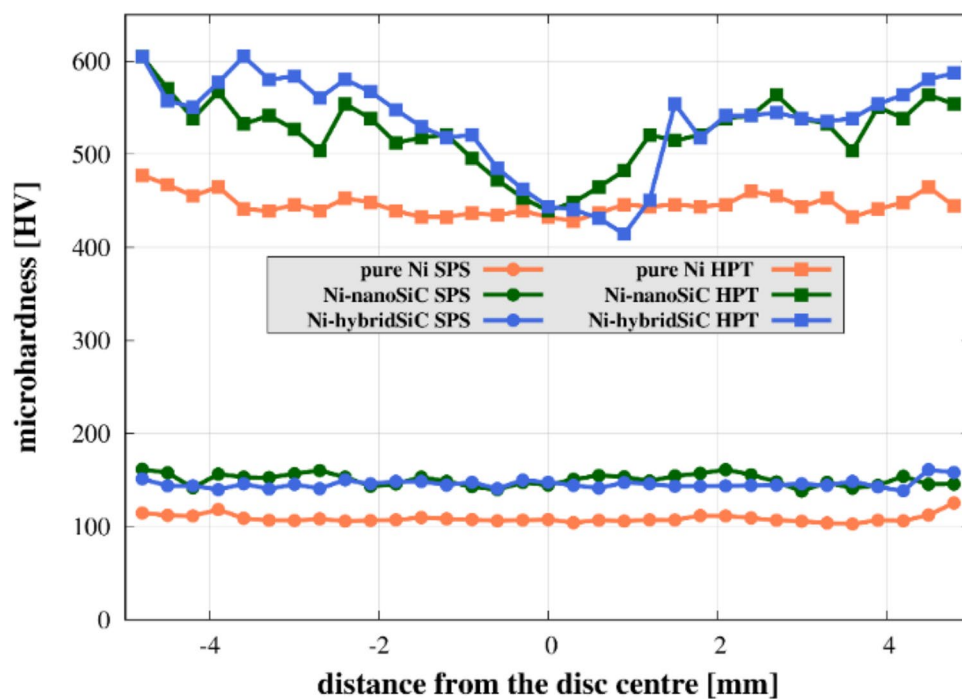


Figure 8 Summary of microhardness **a** and tensile strength **b** of Ni and Ni-SiC composites.

Figure 9 Hardness distribution as a function of the distance from the center of the disc.



steep, gradient is observed for the Ni-nanoSiC system, indicating effective strain-induced strengthening enhanced by particle reinforcement. In contrast, SPS-only samples exhibit relatively uniform local hardness distributions across the radial direction, confirming the absence of strain-driven microstructural refinement.

To reduce high internal stresses and decrease the material's brittleness, samples containing SiC nanoparticles and SiC hybrid particles were additionally annealed after HPT. Figure 10 shows the average microhardness after heat treatment. The annealing response of HPT-processed Ni-SiC composites exhibits a non-monotonic temperature dependence, with initial strengthening followed by progressive softening at higher temperatures. Both Ni-nanoSiC and Ni-hybridSiC samples show a significant increase in microhardness after annealing at 200–300 °C. Since the microstructures annealed within this temperature range did not reveal pronounced morphological changes relative to the HPT-processed condition [45], the observed hardening is unlikely to be associated with major microstructural coarsening or morphological transformation. Instead, it may be attributed to more subtle changes within the highly defected HPT microstructure, consistent with recovery-driven rearrangement of dislocations and preservation of the ultrafine-grained structure. This interpretation is further supported by the XRD analysis and the corresponding crystallite sizes (Figure 7b).

The maximum average hardness after annealing depends on the reinforcement architecture. For Ni-nanoSiC, the average microhardness reaches its maximum at 200 °C and subsequently decreases at 300 °C. In contrast, the Ni-hybridSiC composite exhibits further hardening between 200 and 300 °C and reaches its maximum average hardness of approximately 720 HV at 300 °C. At higher annealing temperatures (≥ 400 °C), a pronounced decrease in hardness occurs in both systems, attributable to grain growth and reduced dislocation density. This softening effect becomes especially severe above 500 °C, where the microhardness drops below 300 HV for both materials due to the recrystallization process and intensive grain growth occurring. This reduces the number of grain boundaries, which act as barriers to dislocation motion, thereby reducing the material's thermal stability and mechanical properties.

After annealing, the hardness distribution for Ni-nanoSiC composites (Figure 11a) still reflects the characteristic radial gradient induced by HPT, with lower values near the disk center and higher hardness toward the edges, particularly at 200–300 °C. The maximum average hardness is observed at 300 °C, where values locally approach ~850–900 HV at larger radii. Increasing the annealing temperature (≥ 400 °C) progressively reduces both the overall average hardness and the radial gradient, leading to a more homogeneous but softer response, especially at 600–700 °C.

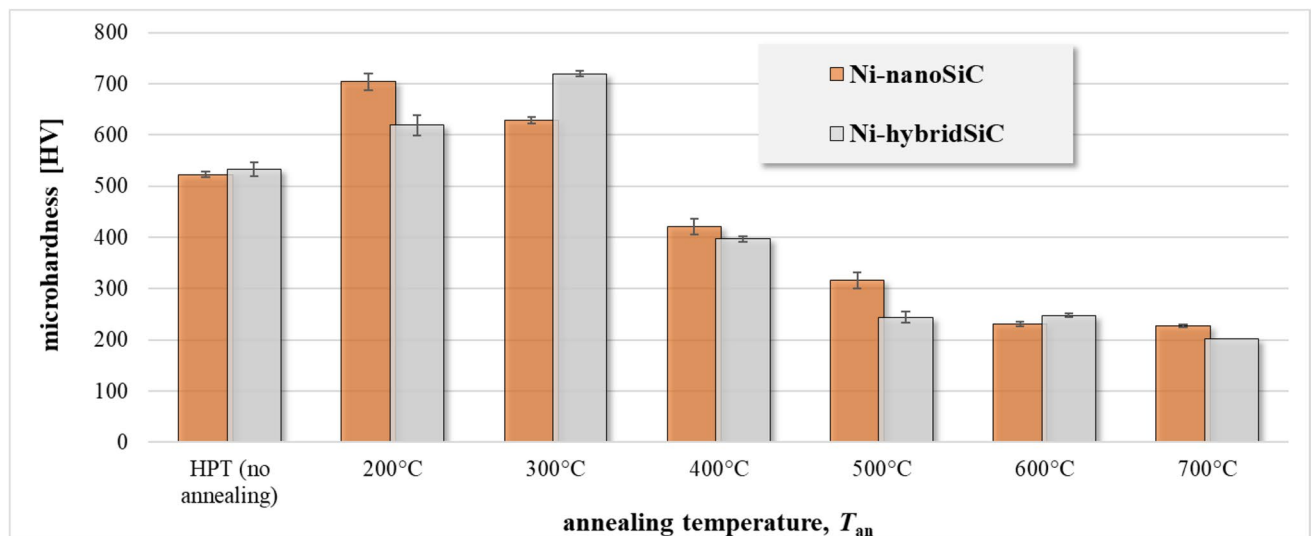


Figure 10 Average microhardness results after annealing treatment.

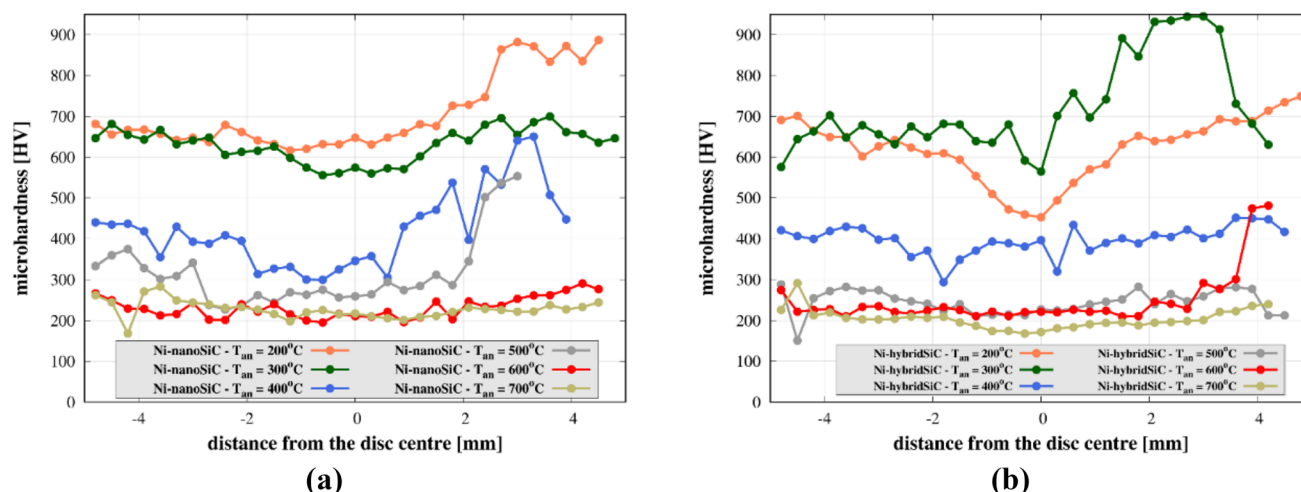


Figure 11 Hardness distribution as a function of the distance from the center of the disk after annealing treatment for Ni-SiC composites with: **a** nanosized and **b** hybrid ceramic reinforcement.

A similar trend is observed for the Ni-hybridSiC system (Figure 11b), although the radial variation is more pronounced, with peak hardness (local radial maximum) exceeding ~ 900 HV at ~ 2 – 3 mm from the center after annealing at 300°C . This suggests a stronger synergistic effect of bimodal reinforcement and strain accumulation at intermediate radii. At higher annealing temperatures, the hardness decreases and the distribution becomes flatter.

Tensile properties

The tensile response of Ni-SiC composites is strongly dependent on both the processing route and the type of ceramic reinforcement. SPS-processed samples show relatively low tensile strength but maintain considerable ductility, reflecting a coarse-grained microstructure with limited strain hardening. Pure Ni after SPS reaches 411 ± 2 MPa with a high elongation of $\sim 36\%$, which serves as a reference for ductile behavior. The addition of nanosized SiC increases the tensile strength to 569 ± 13 MPa, but significantly reduces elongation to $\sim 6.5\%$. The lower elongation may be due to a greater number of particles, indicating the onset of particle-induced strain localization and consequently, more potential sites for crack initiation. In contrast, the hybrid SiC system retains high ductility ($\sim 29.5\%$) while achieving a comparable strength level of 556 ± 3 MPa, suggesting a more favorable balance between reinforcement and matrix plasticity. This behavior suggests that the bimodal

particle distribution mitigates stress-concentration effects and delays crack initiation.

The application of HPT results in a substantial increase in tensile strength due to severe plastic deformation, grain refinement, and a high dislocation density. For pure Ni, the tensile strength increases more than threefold to 1367 ± 8 MPa, although elongation drops sharply to $\sim 2.8\%$. A similar strengthening trend is observed for Ni-nanoSiC, with strength reaching 1001 ± 61 MPa; however, ductility is drastically reduced to $\sim 0.19\%$. This extremely low elongation indicates a transition to quasi-brittle behavior, where plastic deformation is highly constrained (Figure 12). The Ni-hybridSiC composite processed by HPT achieves an even higher tensile strength of 1454 ± 79 MPa, confirming the superior strengthening efficiency of the hybrid reinforcement. Despite this, elongation remains very limited ($\sim 0.62\%$), reflecting the dominant role of strain localization and early crack propagation. The stress-strain curves clearly show a steep elastic-plastic transition followed by premature failure, characteristic of ultrafine-grained materials.

Annealing at 300°C further modifies the mechanical response by enabling partial recovery of the highly deformed microstructure. In the case of Ni-nanoSiC, the tensile strength increases to 1390 ± 42 MPa, exceeding the HPT-only condition, which suggests stabilization of dislocation structures and possible relaxation of internal stresses. The elongation slightly improves to $\sim 0.39\%$, although it

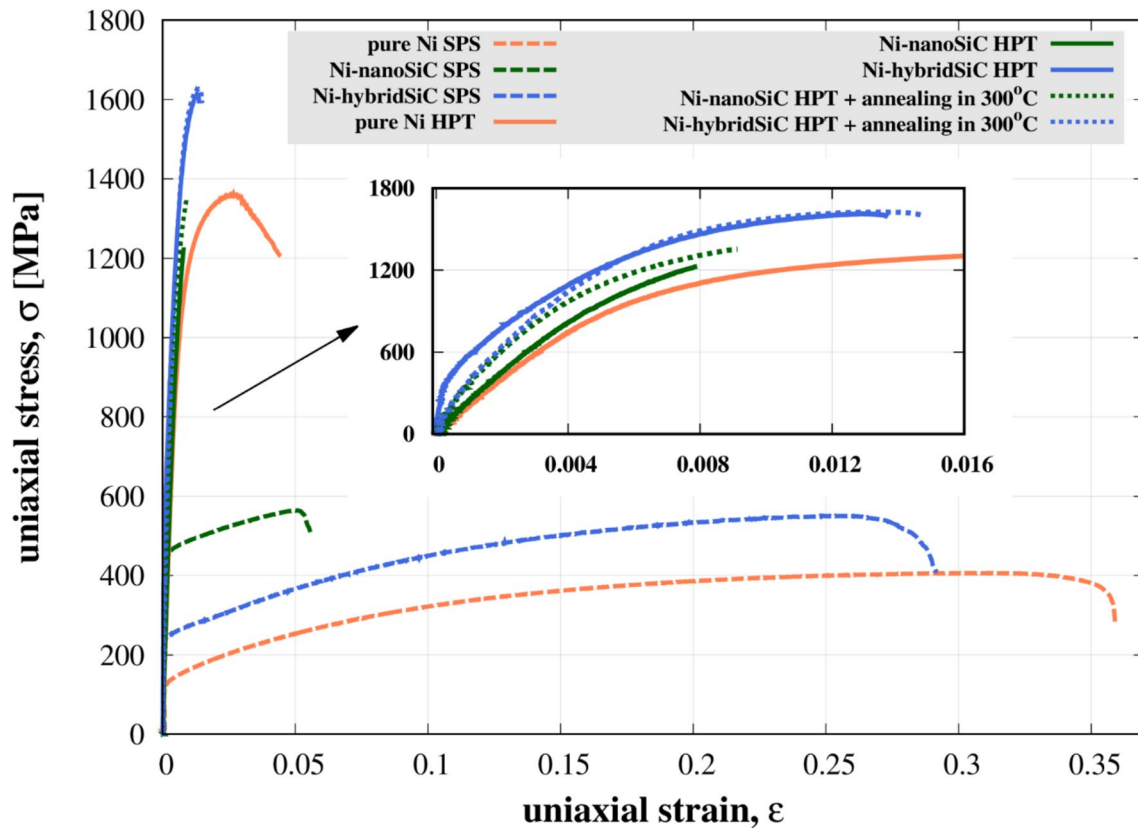


Figure 12 Representative stress–strain curves of pure Ni and Ni–SiC composites processed by SPS, HPT, and HPT+annealing at 300 °C.

remains extremely low, indicating limited ductility recovery. For the Ni–hybridSiC composite, annealing yields the highest tensile strength across all conditions, reaching 1526 ± 18 MPa. This confirms that the hybrid system benefits most from combining HPT with post-deformation heat treatment. The corresponding elongation ($\sim 0.6\%$) shows only a marginal change compared to the HPT state, indicating that strengthening mechanisms dominate over any ductility recovery. The failure elongation values closely follow the ultimate elongation trends, confirming the absence of significant post-necking deformation.

Discussion

The effect of SPS processing

The spark plasma sintering process appears to have produced a highly densified microstructure of Ni-based composites (Figure 3a). Since relative density measurements indicate a residual porosity of around

2–3% (Table 1), no pores are visible, even those near reinforcement particles. The Ni matrix appears to form a homogeneous domain with coarse, equiaxed grains and annealing twins (Figure 4a and b). The interfaces between the Ni matrix and both micro- and nanoscale SiC particles appear well bonded, indicating effective particle–matrix consolidation under the combined influence of pulsed electric current, pressure, and rapid heating characteristic of SPS processing. The homogeneity of the microstructure in high-energy-milled and subsequently sintered samples was demonstrated by the microhardness distribution (Figure 9). Regardless of the material studied—pure Ni or Ni–SiC composites—very similar HV results were obtained along the sample diameter. The addition of SiC particles resulted in a $\sim 50\%$ increase in microhardness relative to pure Ni (Figure 8a). A larger increase ($\sim 100\%$) was reported in [27], as similar SPSed Ni-3% vol. SiC nanocomposites were investigated.

The increase in microhardness, as compared to pure Ni and Ni–SiC SPSed composites, is clearly associated with several strengthening mechanisms. Firstly, the

Table 1 Density results of nickel-based materials

Material	Theoretical density, ρ_t [g/cm ³]	Measured density, ρ_m [g/cm ³]	Relative density, ρ_{rel} [%]
Pure nickel SPS	8.90	8.70	97.8
Pure nickel HPT		8.87	99.6
Ni-nanoSiC SPS	8.73	8.44	96.7
Ni-hybridSiC SPS		8.56	98.0
Ni-nanoSiC HPT		8.69	99.5
Ni-hybridSiC HPT		8.70	99.7
Ni-nanoSiC HPT + annealing (300 °C)		8.60	98.5
Ni-hybridSiC HPT + annealing (300 °C)		8.64	99.0

addition of ceramic particles to a nickel matrix leads to a significant increase in hardness via dispersion strengthening, in which hard, non-deformable particles impede dislocation motion (Orowan mechanism) [46]. Furthermore, the reinforcing particles restrict grain-boundary migration through Zener pinning [45], thereby suppressing grain growth and stabilizing a finer matrix grain structure with a higher grain-boundary density, which contributes to hardness enhancement according to the Hall–Petch relationship [47]. Finally, the application of high-energy milling before SPS promotes intensive plastic deformation and repeated fracture–welding of powder particles [48], resulting in a more refined and defect-rich microstructure (Figure 4b). As a consequence, the SPSed Ni-hybridSiC composite exhibits a substantial reduction in average grain area by approximately 56%, consistent with the observed shift toward smaller equivalent grain diameters (Figure 4e and f).

As shown, the grain size depends significantly on composite composition and manufacturing conditions [49]. Applied sintering process parameters (sintering temperature: 1000 °C, sintering/ d_{well} time: 10 min, external pressure: 50 MPa) were selected based on our previous papers on micro-sized SiC particles reinforced with a relatively large ceramic content (20% vol.) [7, 16]. In this work, a pronounced microstructural transformation occurs, associated with the partial or even complete dissolution of SiC, resulting from an intensive reaction between nickel and silicon. This process leads to the formation of a complex interphase composed of a nickel silicide matrix ($Ni_{31}Si_{12}$ and/or Ni_3Si) enriched with carbon as nanoscale precipitates. The phase transformation in the Ni–Si system has been confirmed in several studies involving heat treatment at elevated temperatures (over 800 °C) [50, 51].

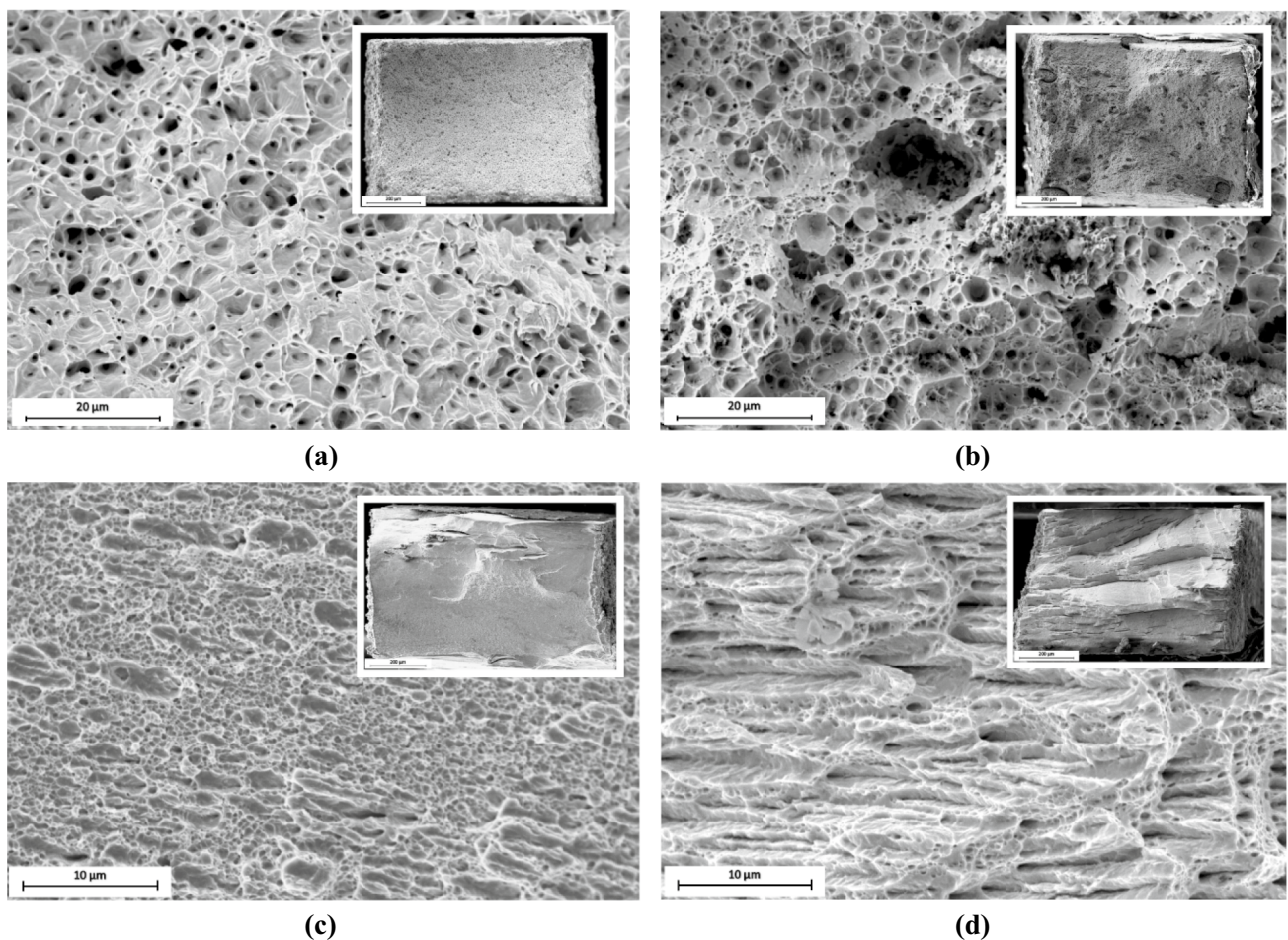
A similar effect can be observed in Figure 5, where nanosized particles are saturated with both Ni and Si. Moreover, as shown in [16], with increasing thermal exposure, carbon nanoprecipitates undergo coarsening and agglomeration, evolving into larger carbon particles. Such carbon segregation is evident in Figure 5, where several spots in the composite microstructure are saturated with carbon and contain no nickel or silicon. A more detailed analysis of the microstructural evolution of Ni–SiC composites manufactured by the SPS method can be found in [7].

As the tensile properties were not improved (relative to pure Ni) in our previous paper [15] due to the large amount of ceramic reinforcement, the addition of 3% vol. SiC nano/hybrid SiC particles here enhanced the effective properties considerably. The tensile strength of Ni–SiC composites (irrespective of reinforcement size) grows from ~400 MPa (pure Ni) to ~550–600 MPa (Figure 12), which is quite similar to the result of corresponding composites studied in several papers [28, 29, 52, 53]. The failure elongation results reported in the cited investigations fall between our nano- (~6.5%) and hybrid-type (~30%) SPS samples (Table 2). Basically, they can be placed around 10–15% depending on milling time [28]. As shown in [28], annealing improves the ductility of sintered Ni–SiC composites by a few percent. In turn, the strain-to-failure during micro-testing of nickel-based composites with nano-SiC [52] provides relatively good agreement with failure elongation of our Ni-nanoSiC SPS (~7–9.5% vs ~6.5%).

The similarity in tensile properties extends to the fracture characteristics of the studied materials (Figure 13). As confirmed in the literature [54, 55], the fracture surface of the pure Ni SPS sample exhibits a classic ductile morphology, characterized by a

Table 2 Average results (with standard error) of the microhardness and tensile tests of Ni–SiC composites

Material	Micro-hardness [HV]	Tensile strength, σ_{UTS} [MPa]	Ultimate elongation, ε_u [%]	Failure elongation, ε_T [%]
Pure Ni SPS	108	411 ± 2	35.62 ± 0.61	36 ± 0.73
Ni-nanoSiC SPS	150 ± 1	569 ± 13	6.46 ± 1.85	6.54 ± 1.85
Ni-hybridSiC SPS	145	556 ± 3	29.43 ± 0.52	29.49 ± 0.53
Pure Ni HPT	445 ± 2	1367 ± 8	2.84 ± 0.53	3.23 ± 0.63
Ni-nanoSiC HPT	523 ± 6	1001 ± 61	0.19 ± 0.04	0.51 ± 0.06
Ni-hybridSiC HPT	533 ± 9	1454 ± 79	0.62 ± 0.33	0.72 ± 0.38
Ni-nanoSiC HPT + annealing (300 °C)	629 ± 7	1390 ± 42	0.39 ± 0.04	0.39 ± 0.04
Ni-hybridSiC HPT + annealing (300 °C)	720 ± 20	1526 ± 18	0.59 ± 0.06	0.63 ± 0.07

**Figure 13** SEM images of the fracture surface of: **a** Ni SPS, **b** Ni-hybridSiC SPS, **c** Ni HPT, and **d** Ni-hybridSiC HPT.

homogeneous distribution of equiaxed dimples, indicating that microvoid nucleation, growth, and coalescence are the dominant failure mechanisms (Figure 13a). In contrast, the Ni–SiC SPS sample shows a more heterogeneous fracture surface with a mixed

ductile–brittle character. While dimples are still present, they are less uniform and often interrupted by larger voids, particle pull-out sites, and irregular cavities associated with the presence of SiC reinforcements [52]. Metal–ceramic interfaces exhibit reduced

interfacial cohesion and are therefore more prone to failure under tensile loading, which likely accounts for the observed presence of SiC nanoparticles embedded within the dimples [29].

The effect of HPT processing

In nickel, which has an FCC crystal structure with a high number of active slip systems, grain refinement during HPT initiates with plastic deformation dominated by dislocation slip within initially coarse, equiaxed grains [42]. Progressive strain leads to intense dislocation accumulation and interaction, resulting in the formation of subgrain structures that evolve into low-angle grain boundaries (LAGBs) as misorientation increases. With continued deformation, these subgrains are progressively refined into ultrafine grains, while increasing lattice rotations promote the transition to higher-angle boundaries [43]. Ultimately, a steady-state microstructure is reached (Figure 3b), in which dislocation generation and annihilation at grain boundaries are balanced, yielding a stable, highly refined, elongated-grain structure characteristic of severely deformed nickel (Figure 4cd). The grain size distribution of pure Ni (Figure 4g) confirms a significant shift toward submicron dimensions after HPT, with an average grain size of approximately 0.3 μm .

Several studies report similar values for pure Ni after HPT, depending on the number of turns/revolutions/rotations. At strains greater than 0.5 rotation (under an applied pressure of 4 GPa), the grain size distributions (with an average diameter of 0.3–0.5 μm) show no significant change, thereby indicating saturation of the grain-refinement process [42]. The same effects have been achieved with 5–10 turns, yielding an average grain size of 0.21–0.33 μm , depending on the specimen spot [43]. Higher revolutions result in greater microstructural homogeneity [56], as evidenced by a relatively flat microhardness distribution across the sample diameter (Figure 9). However, as stated in [57], pure metals do not undergo further grain refinement once the steady state is reached due to stress-induced grain-boundary migration, with the saturation grain size in plastically deformed Ni estimated at approximately 200 nm, which is consistent with our studies.

The presence of SiC particles introduces a dual-microstructure effect. On the one hand, it enhances grain fragmentation, decreasing the average grain size to $\sim 0.2 \mu\text{m}$. Since there are no reports on HPTed

Ni-SiC composites, we can find an analogy to HPTed Ni composites with carbon nanotubes [41]. There, Ni-3wt.%CNT composites indicate a mean grain size of around 0.25 μm . A similar effect was achieved in [58]. XRD studies have also confirmed the improved fragmentation of the Ni matrix upon addition of SiC particles, as the average crystallite size decreases by 14–31% depending on the peak considered (Figure 7).

On the other hand, SiC particles reinforcing the Ni matrix processed by HPT exhibit greater heterogeneity, as evidenced by microhardness distribution (Figure 9). The results clearly demonstrate that the radial variation in hardness reflects the non-uniform plastic deformation, with maximum strengthening occurring at the disk periphery. The characteristic “U-shaped” microhardness distribution observed in nickel HPT-processed samples is typical and reflects the absence of a steady-state saturation at low numbers of revolutions [42, 43]. Nickel composites with nanotubes [41] bring a similar distribution after HPT as those with SiC particles. While both Ni-SiC and Ni-CNT systems exhibit the characteristic HPT-induced radial hardening due to strain gradients, the Ni-SiC composites—particularly with hybrid reinforcement—demonstrate a more homogeneous microhardness distribution across the disc, whereas CNT-reinforced Ni shows a stronger center–edge gradient despite achieving slightly higher peak hardness, especially due to the presence of ceramic clusters and agglomerates [59]. In our studies, the fragmentation of micro-SiC and the redistribution of nano-SiC indicate effective particle refinement and homogenization (Figure 3b). SiC reinforcement exhibits a bimodal size distribution, with finely dispersed nanoparticles ($\sim 50 \text{ nm}$) and fragmented, elongated microparticles ($\sim 0.5 \mu\text{m}$), similar to carbon nanotubes after HPT [60]. TEM-EDS analysis further revealed distinct chemical characteristics of the two reinforcement populations. The fragmented microparticles retained a C-rich quasi-fiber structure (Figure 6), suggesting partial decomposition and elemental redistribution during processing. Moreover, TEM/EDS analysis revealed a pronounced co-localization of Ni and Si in regions associated with the nanosized reinforcement, indicating an interaction between the Ni matrix and SiC and the formation of Ni-Si-rich reaction regions. However, the EDS results alone do not allow an unambiguous crystallographic identification of a specific nickel silicide phase. XRD patterns of the HPT-processed composites show only the characteristic reflections of FCC Ni, with no

additional phases detected (Figure 7a). This suggests that the Ni–Si-rich reaction products observed locally by TEM/EDS are not present in a form or amount sufficient to produce detectable reflections in the present XRD measurements.

The evolution of the microstructure of the HPT samples significantly influences their mechanical properties (Figure 8). The application of HPT results in a substantial increase in hardness (~ 445 – 533 HV), driven by severe grain refinement (Hall–Petch strengthening) [61] and strain hardening [62, 63], with a more pronounced effect in SiC-reinforced systems due to enhanced dispersion strengthening (Orowan strengthening) [64] and load transfer. HV results are in good agreement with the literature data. As shown in [43], the nickel sample demonstrates a saturation effect (in the function of torsional strain) at a level close to 400 HV. In [41], severe plastic deformation by HPT with 4 rotations at 4 GPa pressure yields nickel samples with an average microhardness close to 450 HV, which is in good agreement with our results for pure Ni HPT (445 HV).

The increase in microhardness after HPT is partly attributed to significant densification, which effectively reduces residual porosity, as evidenced by the relative density measurements in Table 1. Moreover, the radial microhardness distribution indirectly indicates spatial heterogeneity in the HPT-induced microstructure, which is also relevant to interpreting the tensile results. As the gage section spans a substantial part of the disk diameter, it includes different local HPT-induced microstructural states. Thus, the tensile properties represent an integrated response of the radially heterogeneous microstructure rather than that of a single radial position. Consequently, HPT, compared to SPS, markedly enhances tensile strength (Figure 12), reaching $\sim 3.3 \times$ for pure Ni ($411 \rightarrow 1367$ MPa), $\sim 1.8 \times$ for Ni–nanoSiC ($569 \rightarrow 1001$ MPa), and $\sim 2.6 \times$ for Ni–hybridSiC ($556 \rightarrow 1454$ MPa); however, it leads to a drastic reduction in elongation for all materials compared to SPS, reflecting a pronounced strength–ductility trade-off. A similar character of pure Ni HPT was reported in [65], with a relatively high UTS of 1270 MPa and a reduced elongation at fracture of 6%. In turn, the limited tensile strength of pure Ni was registered in [66], where different states of Ni samples (pure, impurity-containing, carbon-doped) were studied. Since pure Ni HPT yields a 1000 MPa UTS, the presence of impurities/carbon allows for a ~ 40 – 70% stronger

material, which is close to our results for composites with hybrid-sized SiC particles. The highest reported tensile strength was obtained in [67], where Ni/CNT composites at different HPT deformation temperatures were analyzed. The composite with 2% ceramic CNT reinforcement, deformed at 200 °C, achieves a UTS of over 2000 MPa. As confirmed, the strengthening effect of HPT is most pronounced in the composite variant of Ni samples, indicating a synergistic contribution of severe plastic deformation and hybrid particle reinforcement to the load-bearing capacity.

The fracture behavior of HPT can be compared to that of SPSed samples, as shown in Figure 13cd. Since the fracture surface of pure Ni processed by HPT exhibits a fine dimpled morphology, indicating ductile microvoid coalescence, it can be defined as quasi-ductile/semi-brittle fracture [67]. The dimples are significantly smaller and more densely distributed than under SPS conditions, reflecting an ultrafine-grained structure and limited capacity for plastic deformation. In contrast, the Ni–hybridSiC HPT sample exhibits a highly anisotropic, lamellar fracture surface aligned with the shear direction imposed during HPT. This morphology is characterized by elongated dimples, shear bands, and localized cleavage-like facets, indicating strong strain localization and a transition toward mixed ductile–brittle behavior. The presence of reinforcement particles promotes interfacial decohesion and crack deflection, further accelerating damage evolution. The inset figure of Ni–hybridSiC HPT closely resembles the fracture surface of Ni–C composites [66] with a relatively flat fracture surface with sharp edges and limited macroscopic deformation, indicating a predominantly brittle (or quasi-brittle) fracture. The absence of pronounced necking and the planar character of the fracture surface suggest rapid crack propagation with minimal plastic accommodation. This observation is consistent with the very low elongation values measured for the hybrid composite after HPT.

The present fractographic assessment is qualitative and is used to support the interpretation of the tensile response rather than to provide statistically representative fracture-surface metrics. Nevertheless, the transition from a fine dimpled morphology in HPT-processed pure Ni to an anisotropic lamellar surface containing elongated dimples, shear bands, cleavage-like facets, and indications of particle-related interfacial decohesion in Ni–hybridSiC is consistent

with the measured reduction in tensile ductility and the transition toward mixed ductile–brittle fracture.

The effect of annealing treatment

Post-deformation annealing was applied to HPT-processed Ni–SiC composites to relieve internal stresses and mitigate their pronounced brittleness, as has been performed in many recent studies on the annealing of HPT MMCs [68, 69]. The most efficient and straightforward indicator of the annealing effect on mechanical properties is microhardness testing (see Figure 10). An initial increase in hardness up to 200–300 °C is observed in both Ni–nanoSiC and Ni–hybridSiC systems, which can be attributed to recovery processes that reduce dislocation density while preserving the ultrafine-grained framework [70]. SEM observations of the annealed samples (up to 300 °C) did not reveal pronounced morphological changes compared with the HPT-processed condition. A similar behavior has been reported for HPT-processed Ni, for which annealing at 350 °C for 1 h resulted in insignificant changes in the observed microstructure [45]. Therefore, in the present study, the more subtle structural evolution induced by annealing was evaluated by XRD analysis based on changes in crystallite size. As shown in Figure 7, with increasing annealing temperature (300–400 °C), XRD diffraction peaks become progressively sharper, reflecting crystallite growth driven by recovery and the onset of recrystallization [71]. This coarsening is more pronounced at 400 °C, where thermally activated processes significantly accelerate grain growth [72]. It is consistent with the literature [68], which reports that carbon-doped Ni HPT maintains high, nearly stable hardness up to an annealing temperature of ~ 300 °C, indicating strong thermal stability. In contrast, high-purity Ni HPT remains relatively stable up to ~ 150 °C, but after that, it exhibits a sharp drop in hardness. Analogous observations were reported in [71], where a pronounced reduction in the strength of pure Ni HPT within the annealing temperature range ~ 250–420 °C was associated with a substantial increase in crystallite size, accompanied by extensive recovery and annihilation of dislocations forming the HPT-induced crystallite boundaries. On the other hand, annealing HPTed Ni–CNT composites at 300 °C for 3 h results in a noticeable reduction in hardness, with the same evolution observed throughout the sample radius [41]. The hardness was reduced to 48%, 36%, and 24% for pure Ni, Ni-1 wt.% CNT, and Ni-3 wt.%

CNT, respectively. However, as the authors state, the final results may be affected by HPT parameters, since the saturation point after 4 HPT rotations was not reached (even at the sample edges).

For our Ni–hybridSiC composites, the maximum strengthening occurs at ~ 300 °C, particularly in the hybrid composite (~ 720 HV). Such an enhancement is evident in the increased tensile strength of both Ni–SiC variants (Figure 8). As stated in [72], the strength of Ni-based HPT materials arises from the agglomeration and annihilation of vacancies at homologous temperatures between ~ 150 and 250 °C, corresponding to classical annealing stages III and IV. Moreover, annealing of HPT MMC may improve mechanical connectivity at the interface [73].

Noticed an increase in tensile strength occurred despite a slight decrease in relative density after annealing compared to the HPT state (Table 1), which is mainly associated with recovery and stress relaxation, inducing minor volumetric expansion and stabilization of residual microvoids. Additionally, grain-boundary migration and interfacial relaxation in Ni–SiC composites may promote the formation or reopening of nanoscale porosity, which can indicate poor ductility. Compared to HPT samples, the ductility remains relatively unchanged due to a very high density of grain boundaries, residual defects, and reinforcement/matrix interfaces, which act as preferential sites for strain localization and crack initiation [32].

Conclusions

The presented work can be summarized in the following remarks:

- High-energy ball milling followed by spark plasma sintering enabled the fabrication of highly densified Ni–SiC composites with relative densities exceeding 98%, homogeneous reinforcement distribution, and continuous particle–matrix bonding. The addition of SiC particles refined the Ni matrix grain structure and improved both hardness and tensile strength through dispersion strengthening and grain-boundary pinning mechanisms.
- TEM-EDS investigations revealed significant microstructural evolution during SPS processing. Microsized SiC particles exhibited a Si-rich core surrounded by a carbon-enriched shell. In contrast, nanosized particles exhibited simultaneous enrich-

ment in Ni and Si, indicating interfacial reactions and the formation of Ni–Si-rich reaction regions during sintering.

- High-pressure torsion produced an ultrafine-grained microstructure with the average grain size reduced to approximately 0.2–0.3 μm . At the same time, carbon-rich particles underwent fragmentation and elongation along the shear direction, whereas nanosized particles became more uniformly distributed within the matrix.
- The combined effects of severe grain refinement, strain hardening, dispersion strengthening, and enhanced densification resulted in a substantial increase in mechanical performance after HPT processing. Tensile strength increased from approximately 400–600 MPa in SPS samples to 1000–1450 MPa after HPT, while average microhardness increased to 445–533 HV depending on the reinforcement type. This strengthening was accompanied by a drastic reduction in tensile elongation, demonstrating a pronounced strength–ductility trade-off.
- Hybrid reinforcement composed of micro- and nanosized SiC particles provided the most effective strengthening response. The Ni–hybridSiC composite exhibited the highest tensile strength after HPT processing (1454 MPa), demonstrating a synergistic effect between bimodal reinforcement and severe plastic deformation.
- Post-deformation annealing resulted in a non-monotonic evolution of hardness, with maximum strengthening observed at 300 °C. The highest average microhardness (~ 720 HV) and tensile strength (~ 1500 MPa) were obtained for the annealed Ni–hybridSiC composite, however, its ultimate elongation remained very limited, indicating that the applied processing route primarily promotes ultra-high strength and hardness rather than a balanced strength–ductility response.

Declaration of generative AI and AI-assisted technologies in the writing process

During the preparation of this work, the authors used Grammarly and ChatGPT (by OpenAI) to correct spelling and English grammar and to improve the clarity of the images. After using this tool/service, the authors

reviewed and edited the content as needed and take full responsibility for the publication's content.

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